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The main objective of this project is to understand the nature of adsorption, dissociation, reaction, and desorption on semiconductor surfaces when molecules are accelerated to enhance their translational energy in a supersonic beam and when the surface is irradiated by photons. These chemical processes are important for providing a scientific basis for understanding chemical vapor deposition and atomic layer epitaxy of thin films. Special emphasis was placed in understanding carbon-containing molecules in order to search for suitable precursors for the growth of silicon carbide and diamond. The understanding, experience, and knowledge obtained in this study were instrumental in advancing a novel thin film growth technique based on supersonic jet epitaxy, which was successfully transferred to a small high technology company.

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Report #4

IN-SITU ELECTRON AND OPTICAL SPECTROSCOPIES OF TRANSLATIONAL AND VIBRATIONAL ACTIVATED BOND BREAKING AND FORMATION ON SEMICONDUCTORS

Grant No. F49620-92-J-0309
Project-Task 2303/BS, 2305/ES

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4 December 1995

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Final Technical Report

In-Situ Electron and Optical Spectroscopies of Translational and Vibrational Activated Bond Breaking and Formation on Semiconductors

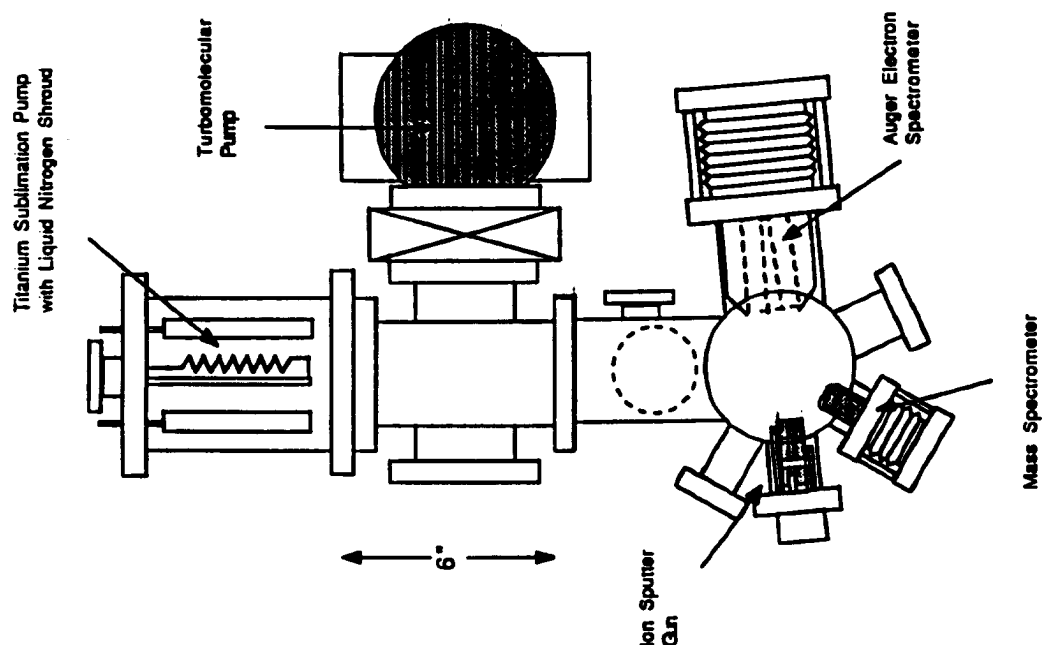**(F49620-92-J-0309, Task 2305/ES)**

III. Objectives:

The main objective of this project is to understand the nature of adsorption, dissociation, reaction, and desorption on semiconductor surfaces when molecules are accelerated to enhance their translational energy in a supersonic beam and when the surface is irradiated by photons. These chemical processes are important for chemical vapor deposition and atomic layer epitaxy of thin films.

IV. Experimental Approach:

The translational energy of the reactants are increased by seeding in H_2 or He and forming a supersonic jet. The nature of the adsorbed species is probed by in-situ, time-resolved electron energy loss spectroscopy and photon-induced desorption. The substrate (Si) is cleaned by ion sputtering and annealing. A schematic of the experimental apparatus is shown in Fig. 1.

V. Executive Summary:

The adsorption and reactivity of the following molecules seeded in a supersonic beam was investigated: CH_xF_{4-x} (x=0-4) $(CH_3)_2N_2$ (azomethane), CH_3X (X=Cl, Br, I), C_2H_2, C_2H_4, C_3H_8, CO_2, N_2O, CO, O_2, and the coadsorption of CO with O created from dissociation of CO_2, N_2O, and O_2. For some systems, direct comparisons of reactivity between Si(100)2x1 and Si(111)7x7 were performed. In addition, the reaction of atomic H with adsorbed CH_3X was determined. Generation of CH_x (x=1-3) fragments for diamond growth, the choice of precursors for diamond and SiC growth, the mechanisms and the role of atomic H in the synthesis of diamond and other thin films, the initial stages of oxidation of Si, and the possible creation of new species and compounds on the surface with supersonic gas jets were the key factors in the choice of the above molecules. Photodesorption of CO from Si(100)2x1 was investigated in detail, providing information into nonequilibrium phenonmena
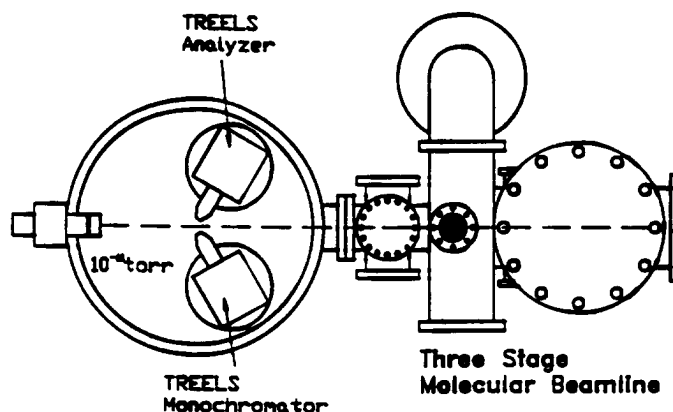
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Fig. 1: Top schematic (top view) shows the upper chamber equipped with surface probes and sputter ion gun. Middle and bottom schematics (top view) show relationship of molecular beam and in-situ time-resolved electron energy loss spectrometer housed in the lower chamber. Sample can be transferred between the two chambers.

In-situ TREELS of Beam-Surface Interactions

TREELS Analyzer

TREELS Monochromator

10^{-4}torr

Three Stage Molecular Beamline

Sample   Apertures   Skimmer   Alumina Nozzle > 1600K

Mass Spectrometer

e⁻

Chopper
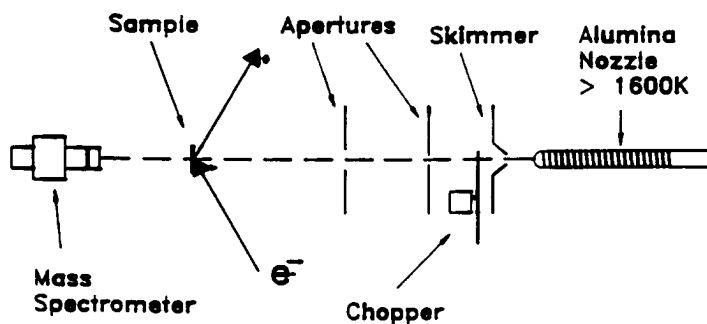
at semiconductor surfaces. Definitive evidence for photodissociation via excitation of surface electronic states of semiconductors was obtained from the photodesorption and photodissociation of H_2S on Si(111). Two graduate students, Rowena Y. Young and Kyle A. Brown, and one postdoctoral associate, Deqing Hu, were involved in the AFOSR supported project during 1992-1995. A total of 7 papers and 1 Ph.D. thesis were published; 3 additional papers are currently in preparation.

VI. Summary of Results of Completed Work

A. Fluoromethanes and Unsaturated Hydrocarbons

A series of molecules was investigated as potential precursors of carbon source, specifically for the synthesis of diamond and SiC. We have used absolute sticking coefficient measurements to determine the reactivity of the fluoromethane series (CH_xF_{4-x} x=0-4), alkanes (C_2H_6, C_3H_8, C_4H_{10}), and alkenes (C_2H_4), and alkynes (C_2H_2). A schematic of these molecules is given in Fig. 2. In Table I, the initial adsorption coefficient (S_0) of the fluoromethane series on Si(100)2x1 is summarized. Similar studies established that CH_3Cl is as reactive as CH_3F, making it an interesting candidate for chemical vapor deposition of diamond films. If the surface is saturated with H, no adsorption of CH_3Cl was observed. Dosing the partially hydrogenated surface resulted in the coadsorption of preadsorbed H and the dissociated products: CH_3 and Cl.

Both acetylene (C_2H_2) and ethylene (C_2H_4) adsorb and dissociate readily on Si. Ethane (C_2H_6) adsorbs only molecularly at low temperatures and does not dissociate. As the molecules get larger, e.g. propane (C_3H_8) and butane (C_4H_{10}), dissociation occurs as the translational energy is increased, with appreciable dissociation occurring for energies greater than about 0.7 eV.

B. Photon-Induced Desorption and Dissociation

We have investigated the photon-induced processes on Si(100) and Si(111) surfaces, which provide a scientific basis for photochemical processing on semiconductor surfaces. We have found the highest recorded photodesorption cross section for CO on Si(100) compared to all systems studied to-date, including photodesorption from metal surfaces. The photoyield was measured as a function of the wavelength. The results show unambiguously that photodesorption proceeds by a substrate mediated mechanism whereby the incident photons
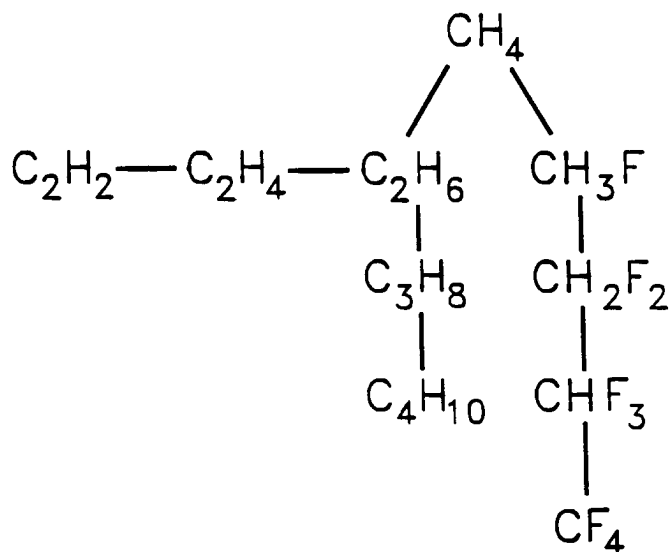
$$CH_4$$

$$C_2H_2 — C_2H_4 — C_2H_6 \quad CH_3F$$

$$C_3H_8 \quad\quad CH_2F_2$$

$$C_4H_{10} \quad CHF_3$$

$$CF_4$$

Fig. 2: Summary of systematic variations from methane as carbon sources for growth of SiC and diamond films.

Sticking Coefficients
of CH_nF_{4-n} (n=0,4) on Si(100)2x1

Molecule	Nozzle Temp(K)	Translational Energy(eV)	S_o
CH_4	1400	1.6	$<10^{-4}$
CH_3F	300 ⋯ 1100	0.1 ⋯ 3.0	≈0.1
CH_3Cl	300	0.1	≈0.1
CH_2F_2	–	–	trapping
CHF_3	800	3.5	$<10^{-4}$
CF_4	1300	4.2	$<10^{-4}$

Table I: Summary of adsorption behavior of CH_xF_{4-x} (x=0-4) on Si(100)2x1. S_o is the initial sticking coefficient.

are absorbed by the substrate and create electron-hole pairs. These excited carriers can migrate to the surface and interact with the Si surface. The excited surface becomes nonbonding to the CO molecules, causing the CO molecules to desorb. In the photodesorption and photodissociation of H_2S on Si(111), the surface electronic states of Si were found to be involved directly in the photon-induced processes.

C. CO_2

For the first time, CO_2 is observed to adsorb on both clean and oxidized (by either ambient O_2 or a hyperthermal N_2O molecular beam) Si(100) surface. At high exposures, multilayer adsorption occurs and desorbs at 85 K. Three characteristic EEL peaks at 84, 168, and 295 meV are observed, which correspond to CO_2 bending, symmetric, and asymmetric vibrations. At surface temperature greater than 80 K, no ambient CO_2 adsorption is observed. However, by increasing the translational energy, hyperthermal CO_2 molecules are found to dissociatively adsorb on Si(100) surface as CO and atomic O in addition to molecular CO_2 adsorption. After a clean Si(100) surface is exposed to 1.0 eV CO_2 molecular beam, four EEL peaks are observed. The 222 meV peak is assigned to C-O stretch, the 98 meV to terminally bonded atomic O , while the 168 and 292 meV to symmetric and asymmetric stretches of CO_2. This assignment is independently confirmed by thermal desorption measurements. The adsorbed CO from CO_2 dissociation is photo-inactive with respect to full-arc light from Hg-Xenon arc lamp.

D. CO

Adsorbed CO from the ambient on Si(100) desorbs at 181 K at low exposures. As CO exposure exceeds 0.1 L, a second desorption peak appears at 163 K. However, only two EEL peaks are observed, which correspond to C-O stretch vibration at 261 meV and Si-CO stretch at 50 meV, respectively. It suggests that CO molecules are terminally bonded on Si(100) surface on electrophilic sites, i.e. buckled-down Si sites, and the second desorption peak at high coverages is due to CO mutual interactions. These head-on bonded CO molecules are photo-active with a high photodesorption yield.

Exposure of Si(100) to a hyperthermal CO molecular beam creates a new CO adsorption state in addition to the ambient adsorbed CO state (see Fig. 3). The ambient adsorbed CO state is found to be the precursor to the new CO state. The activation barrier
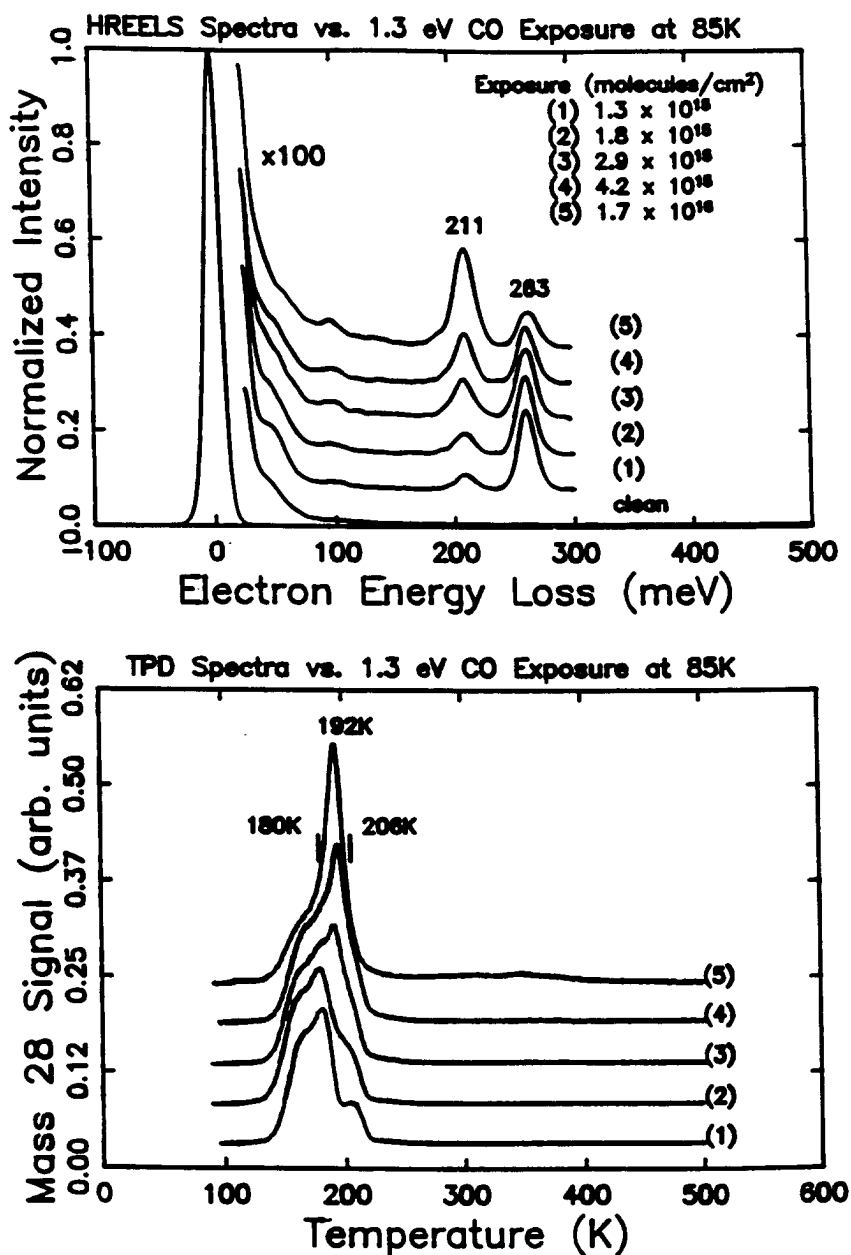
Fig. 3: (Top Spectra) Vibrational spectra for increasing exposures to 1.3 eV CO molecules in a supersonic beam. The two vibrational peaks at 211 meV and 263 meV correspond to C-O stretch attributed to CO bonded in the bridge and terminal sites, respectively. The 211 meV peak is only populated with enhanced translational energy. (Bottom Spectra) CO thermal desorption spectra corresponding to the exposures in the Top Spectra. The 192 K peak is most prominantly associated with the novel CO at 211 meV.

for CO to populate this new state is about 0.9 eV, and normal energy scaling rule is obeyed. From EELS measurements, the C-O vibration (211 meV) in this new state is red-shifted by 50 meV compared to the ambient adsorbed CO (261 meV). It indicates that the C-O bond in this new state is significantly weakened or perturbed. The CO molecule appears to be bridge-bonded between two Si atoms in the dimer through the C (see Fig. 4). However, CO in the new state desorbs at about 200 K, which is only slightly higher than the ambient adsorbed CO. The CO in the new state is photo-active. By comparing this new CO state to the adsorbed CO from CO_2 dissociation, smaller vibrational frequency (211 vs. 222 meV) and lower desorption temperature (200 K vs. 310 to 440 K) indicate coadsorption effect of CO and atomic O (and also probably CO_2). Based on desorption temperatures, CO in the new state is similar to ambient adsorbed CO. On the other hand, it is similar to the adsorbed CO from CO_2 dissociation if vibrational frequency is considered. A full understanding of this seemingly contradictory comparison is anticipated through *ab-initio* density functional calculations which are under way.
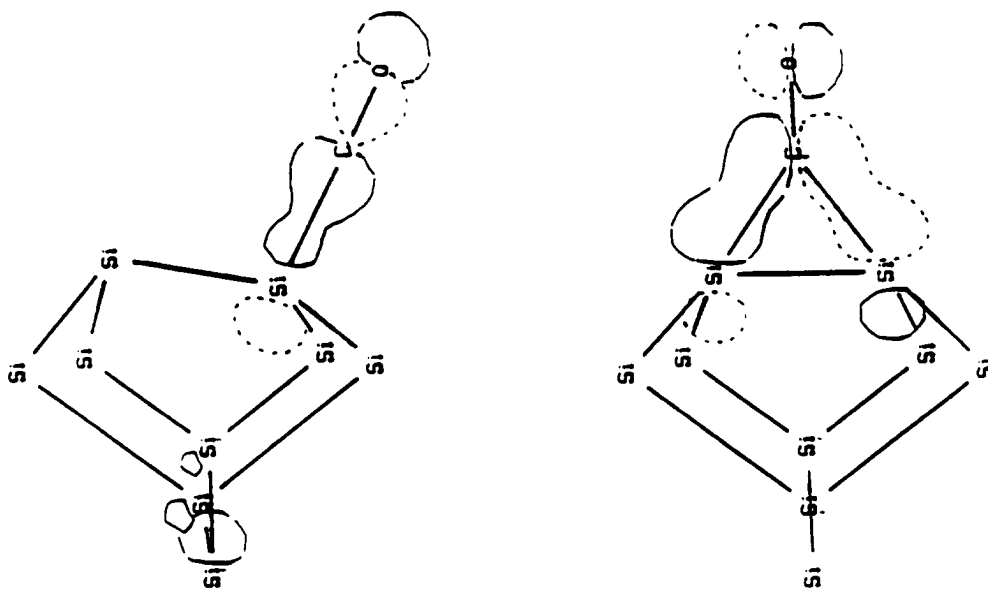

Fig. 4: Bonding geometry of CO as derived from extended Huckle calculations by Qiang Liu and Roald Hoffmann of Cornell University. (Left) CO bonded terminally to an electron deficient site; partial electron is transferred to the other Si of the dimer. (Right) Bridge bonded CO. In both cases, the dimer bond remains intact.

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E. N_2O

N_2O is isoelectronic to CO_2. The dissociative adsorption probability of ambient N_2O on Si(100) surface at room temperature is very small, which has been estimated to be 2.5×10^{-5} by other groups. However, Si surfaces oxidized by N_2O has peculiar properties with respect to either O_2 or H_2O oxidation. First, an oxidized monolayer could be formed by N_2O instead of O_2 or H_2O. Second, the incorporation of a small amount of N in the oxidized layer results in an oxidized layer of better insulating quality compared to bare oxidized layer. It is desirable to have a better understanding of N_2O dissociative adsorption kinetics and dynamics, which is also beneficial to the understanding of CO_2 dissociative adsorption. We found that the initial dissociative adsorption probability of N_2O on Si(100) is increased by 4 orders of magnitude with hyperthermal N_2O beam. The activated dissociative adsorption of N_2O mainly results in the oxidation of Si(100) surface. At a high exposure, a small amount of coadsorbed N_2 and N_2O with O is also observed. In retrospect, it is quite different from the dissociative adsorption of CO_2 on Si(100), which results in comparable amount of coadsorbed CO, O, and CO_2. The activation barrier for N_2O dissociation is lower than CO_2, and the rule of normal energy scaling is also obeyed. Three characteristic EEL peaks at 118-130, 81-89, and 47 meV are attributed to asymmetric, symmetric stretch, and bending vibrations of bridge bonded O on Si(100), respectively. From the EEL spectra alone for the initial stage of oxidation of Si(100), there is no obvious difference between oxidized Si(100) by N_2O and that by ambient O_2 at 85 K.

F. Initial Stage of Oxidation of Si and O + CO

The initial stage of oxidation of Si surfaces has been intensely studied both experimentally and theoretically for a long time because of its technological importance in the electronics industry. However, it is still a debated subject. To our knowledge, this is the first attempt that the initial stage of Si oxidation is investigated through the coadsorption of O and CO on Si(100) at 85 K. This study is also informative to gain a full understanding for the dissociative adsorption of CO_2 and the activated adsorption of CO on Si(100). O could be obtained from either O_2 or N_2O dissociation, and a clean Si(100) surface could be precovered by either O or CO. Therefore, there are in total four different combinations for the coadsorption of O and CO. We have found different reactivities for Si(100) oxidized by O_2

and by N_2O towards CO. First of all, CO-saturated Si(100) surface at 85 K is inert to ambient O_2. After a 100 L ambient O_2 exposure, only a very small amount of O is observed to coexist with CO by both EELS and AES. However, when CO saturated Si(100) surface is exposed to N_2O molecular beam, we found that in addition to adsorbed O, part of ambient adsorbed CO is transformed from the ambient adsorbed state of a $^{12}C^{18}O$ stretch vibration of 252 meV to a new state with $^{12}C^{18}O$ stretch vibration of 216 meV. The transformation process of adsorbed CO from one state to the other is not resulted from N_2O bombardment, but rather from adsorbed O due to N_2O dissociation. Furthermore, ambient CO can adsorb on partially oxidized Si(100) surface by ambient O_2. The O_2 oxidation does not affect the C-O stretch vibration frequency.

It is evident that on N_2O oxidized Si(100) surface $^{12}C^{18}O$ can adsorb in two distinguished states with $^{12}C^{18}O$ stretch vibrational frequencies of 252 and 216 meV, respectively. These coadsorption results strongly suggest that atomic O from N_2O dissociation breaks up the surface dimer bonds and bridges between two Si atoms. The resulting surface Si dangling bonds facilitate CO molecules to form a bridge bonded CO through C with its bond axis perpendicular to the surface. Ambient O_2 adsorbs and dissociates on Si(100) surface, and the resulting O atom has a high probability to go to the backbond sites first before attacking the surface dimer bonds because of the open structure of Si(100) surface. The chemical reactivity of partially O_2 oxidized Si(100) towards CO also supports the observation of coalesced oxidation by STM. The coadsorption of CO and O from N_2O dissociation mimics the coadsorbed CO and O from CO_2 dissociation. In other words, the CO_2 dissociation could be considered to occur in two steps, i.e., atomic O adsorption and then CO adsorption or vice versa. Even though CO_2 and N_2O are isoelectronic, N_2O dissociates into N_2 and O while CO_2 into CO and O. CO and N_2 are also isoelectronic, but CO is much more reactive than N_2. The inertness of N_2 could explain why there is no significant amount of N_2 adsorption occuring on Si(100) as N_2O decomposes.

G. Azomethane $(CH_3)_2N_2$

The applications of diamond thin films in electronic industry require diamond films of a good crystallinity. Heterogeneous growth of single crystalline diamond thin film remains a great challenge for scientists from many different disciplines. To this end, we have

investigated the adsorption of azomethane, methyl halides such as CH_3Cl and CH_3Br on Si(100) surface, and reactions of methyl group and halogen on Si(100) with atomic H. CH_3 radical is believed to be an important precursor for low pressure CVD diamond growth. On the other hand, atomic H plays an indispensable role as an etchant to remove graphitic carbon, abstract surface atomic H, and acts as a surfactant to stabilize metastable diamond nucleation. The reaction between CH_3 radical and atomic H represents a significant kinetic step in the diamond growth process.

Azomethane decomposes into two methyl radicals and one molecular nitrogen. Molecular nitrogen is very inert in general. Therefore azomethane could be used to generate a clean methyl radical beam by pyrolytical decomposition. However, if azomethane decomposes and results in adsorbed methyl radicals on a Si(100) surface with the formation and desorption of molecular nitrogen as ambient azomethane or hyperthermal azomethane beam interacts with Si(100), it would be a more promising precursor for diamond growth. To explore this possibility, we have studied the interaction of Si(100) surface with both ambient azomethane and hyperthermal azomethane molecular beam. It is found that azomethane easily chemisorbs on Si(100) surface at 85 K. Three broad bands are observed in EEL spectra, which can be assigned to C-H stretch at 365 meV, CH_3 deformation at 178 meV, CH_3 wagging and/or N-N stretch at 142 meV, and C-N stretch at 120 meV. Upon annealing, azomethane decomposes with N and C left on the surface to form SiN and SiC. Hyperthermal $(CH_3)_3N_2$ beam does not significantly alter the interaction between azomethane and Si(100) surface.

H. CH$_3$X and H + CH$_3$X

Other good candidates for diamond and SiC thin film growth are methyl halides. The C-X (X = Cl, Br, I) bonds are weaker than C-H bond. The decomposition of methyl halides into methyl radical and halogen is considerably easier than methane which is most often used in the CVD diamond growth process. We found that at higher exposures, both CH_3Cl and CH_3Br form physisorbed multilayer thin films on Si(100) surface at 85 K. CH_3Cl desorbs at 99 K while CH_3Br at 102 K. The observed vibrational spectra for multilayer CH_3Cl and CH_3Br agree with those for free molecular vibrations. Submonolayer of both methyl chloride and methyl bromide easily decomposes into adsorbed CH_3 and Cl or Br on Si(100) surface at

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85 K. The decomposition temperature of adsorbed CH_3 increases with surface coverage up to 847 K. The desorption temperature of halogen on Si(100) surface is above 900 K, which is higher than the decomposition temperature of adsorbed CH_3 on Si(100) surface. However, adsorbed halogen on Si(100) surface can be easily abstracted by atomic hydrogen through Eley-Rideal reaction mechanism. Therefore with a beam of methyl halides and a beam of atomic hydrogen, it is a viable route to synthesize diamond thin films in a high vacuum. A clean growth environment could reduce contamination and lower the concentration of impurities.

The adsorption of atomic H on metal and semiconductor surfaces and the reaction of atomic H with adsorbed species on surfaces are still a topic of intense scientific research because of both fundamental interests and overwhelming technological importance such as diamond growth. Atomic hydrogen is also used as a reagent to clean semiconductor surfaces. With CH_3 adsorbed on Si(100) surface, we have studied the exchange and abstraction reaction of atomic D (deuterium) with H. After the exposure of a CH_3- covered Si(100) surface to a moderate flux of atomic D, both CH_2D and CH_2 species are observed by EELS. The occurrence of CH_2 species on the surface indicates the abstraction of a H atom from CH_3 by D, while CH_2D could indicate the exchange reaction between H and D or two-step reaction involving one H abstraction and further D addition.

I. Cl_2

Dry etching of semiconductor surfaces using Cl_2 has attracted considerable attention recently. As a very effective in-situ cleaning procedure in a high or ultra-high vacuum environment, dry etching technique has a significant technological importance in microelectronics industry. The mechanistic understanding of the adsorption of Cl_2 on Si(100) surface could provide very useful information for optimizing this important dry etching process. For the first time, we observed a multilayer physisorbed Cl_2 on Si(100) surface at 85 K. The multilayer Cl_2 has a characteristic violet color when shining halogen flash light on the surface. Another distinguished feature for multilayer Cl_2 is the strong metallic scattering, resulting in a very weak elastic scattering. The EEL spectrum for such a surface is featureless except for a metallic scattering background. The desorption temperature of the multilayer Cl_2 is about 104 K. After the multilayer Cl_2 desorbs, four EEL peaks at 74, 142,

178, and 213 meV are observed. The 74 meV peak is attributed to Si-Cl stretch of silicon monochloride while the bending mode of Cl is not well resolved. The other three EEL peaks cannot be simply accounted for by advocating the existence of silicon di- or trichloride. By step-wise annealing the surface, these three peaks disappear at temperatures above 146 K. Furthermore, from TPD measurement, a thermal desorption peak at 158 K is observed for mass-to-charge ratio of 98 ($SiCl_2^+$), 63 ($SiCl^+$), 35 (Cl^+), and 28 amu (Si^+ or CO^+). It suggests that silicon dichloride might exist on Si(100) surface after multiayer Cl_2 adsorption. However, the vibrational frequencies of dichloride are too low to account for the EEL peaks.

VII. Development of Human Resources

Two graduate students, Rowena Y. Young and Kyle A. Brown, and one postdoctoral associate, Deqing Hu, were involved in the AFOSR supported project during 1992 - 1995. Deqing Hu holds an NSERC Fellowhip from Canada. Rowena Young is currently in her second year of Law School at Yale University. Kyle Brown just finished his Ph.D. Thesis work (August 1995) and is now on the staff at Applied Materials (Santa Clara, CA). Carsten Rohr is a new student who just joined the group; he will be working with Deqing Hu who will be ending his tenure in December 1995. Another new student will be added to the project in January 1996.

Deqing Hu has begun this summer (1995) to prepare to grow thin films of SiC, AlN, and GaN on 4-inch diameter Si(100) wafers. Simultaneously he is involved in using *ab initio* quantum mechanical pseudopotential method for total-energy calculations of the adsorption of CO on Si(100)2x1. Carsten Rohr will also be involved with thin film growth. Hu and Rohr will continue to carry out fundamental studies of the supersonic molecular beam induced surface chemistry on Si (see Fig. 1).

VIII. Present Progress

During the last year (1994 - 1995), significant amount of data were acquired by Deqing Hu, which have been analyzed and are currently being incorporated into papers. In particular, the following papers are being prepared: 1. adsorption and dissociation of $(CH_3)_2N_2$, 2. the reaction of atomic H with adsorbed CH_3X (X = Cl, Br, I), 3. dissociation of N_2O with enhanced translational energy, 4. comparisons of precursors for SiC and diamond growth: C_2H_2, C_2H_4, C_3H_8, 5. the nature of the initial stages of oxidation of Si(100)2x1 as

revealed by the coadsorption of CO with atomic O formed by the dissociation of CO_2, N_2O, and O_2. The completion of these papers is one of the remaining tasks for Deqing Hu before he departs December 1995.

Deqing has also started *ab initio* quantum mechanical pseudopotential calculations of the total-energy of CO on Si(100)2x1 surface. The objectives are to obtain the different equilibrium binding sites and their total energies. In addition, the translational energy of the incident CO molecules can be varied for investigating the energy barriers and dynamics of adsorption. The occupation of the novel CO binding state as the translational energy increases beyond 0.5 eV will be determined.

We will begin in Fall 1995 the growth of SiC on Si(100) as the first step towards $Si_{1-x-y}Ge_xC_y$ on Si(100). Optical monitoring of Si etching for obtaining ultrathin Si layer in SOI is currently under design. We are also continuing to carry out experiments (Fig. 1) related to the growth and processing of SiC and $Si_{1-x-y}Ge_xC_y$.

IX. List of Publications

1. B.D. Thoms, P.W. Lorraine, and W. Ho, *"A Molecular Beam Study of Ethane on Si(111)7x7: Energy Accommodation and Trapping"*, J. Chem. Phys. **97**, 2759 (1992).

2. D.V. Chakarov and W. Ho, *"Thermal and Photo-Induced Desorption, Dissociation, Reactions of H_2S Adsorbed on Si(111)7x7"*, Surf. Sci. **323**, 57 (1995).

3. R.Y. Young, K.A. Brown, and W. Ho, *"Photodesorption of CO from Si(100)-2x1: Wavelength and Intensity Dependence"*, Surf. Sci. **336**, 85 (1995).

4. K.A. Brown and W. Ho, *"The Interaction of Methyl Chloride and Si(100)2x1"*, Surf. Sci. **338**, 111 (1995).

5. K.A. Brown and W. Ho, *"A Threading Tool for Hard Materials"*, Rev. Sci. Instrum., in press (November, 1995).

6. K.A. Brown, D.Q. Hu, and W. Ho, *"Translationally Activated Dissociation of CO_2 on Si(100)2x1"*, J. Chem. Phys., in press (February, 1996).

7. D.Q. Hu, K.A. Brown, and W. Ho, *"Translational Energy Induced Adsorption of a New CO State on Si(100)-c(4x2)"*, J. Chem. Phys., submitted (1995).

8. K.A. Brown, *"Molecular Beam Induced Surface Reactions and Film Growth"*, Ph.D. Thesis, Cornell University, August 1995.